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- II. Phenacyl, *p*-Chlorophenacyl and *p*-Bromophenacyl Esters of Some Higher Fatty Acids
- III. Isomeric 2-Iminotolyl-3-tolyl-4-*p*-chloro and *p*-Bromophenyl-Delta⁴-thiazolines

DISSERTATION

SUBMITTED TO THE BOARD OF UNIVERSITY STUDIES OF THE
JOHNS HOPKINS UNIVERSITY IN CONFORMITY WITH
THE REQUIREMENTS FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY

By
RAYMOND M. HANN

EASTON, PA.
MACK PRINTING COMPANY
1933

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52, 818 (1930); 55, 4998 (1933).]

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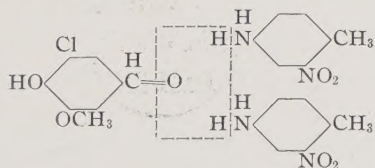


Schiff's Bases Derived from 5-Chlorovanillin*

The intermolecular condensation of aldehydes and amines with subsequent loss of water leads to the formation of the well-known Schiff's bases. Compounds of this class containing the vanillin nucleus as an integral part of their structure have been investigated by a number of workers. Senier and Forster,¹ in carrying out a series of studies upon the thermotropic properties and the effect of actinic light upon Schiff's bases, prepared a series of vanillin amines. Hann² studied the effect of the introduction of iodine into the vanillin residue upon the thermotropic properties of the resulting bases when the iodinated aldehyde was condensed with various amines. Hann and Spencer³ isolated chlorovanillalaniline and chlorovanillal- α -naphthylamine. 2-Bromovanillin and 6-bromovanillin have been condensed with benzidine by Raiford and Stoesser,⁴ and a number of bases from 5-bromovanillin and 5,6-dibromovanillin have been prepared by Raiford and Hilman.⁵

In the present paper is described a further series of compounds of 5-chlorovanillin with aromatic amines. In a majority of cases the reaction products were obtained quite readily in crystalline condition upon cooling an organic solvent containing molecular proportions of the necessary components.

In one instance, that of *m*-toluidine, no crystalline product could be isolated although the marked deepening of the color as the reaction mixture was heated indicated a progressive formation of the chromophore $C=N-$. That this conclusion was warranted was proved by separation of the picrate of 5-chlorovanillal-*m*-toluidine upon addition of alcoholic picric acid. Hantzsch and Schwab⁶ reported derivatives of a type in which one molecule of aldehyde condensed with two molecules of an amine, and in this study such a reaction product was encountered with nitro-*p*-toluidine. The reaction in this case may be considered to proceed in the following manner



* Hann, Jamieson and Reid, *J. Am. Chem. Soc.*, **51**, 2586 (1929).

¹ Senier and Forster, *J. Chem. Soc.*, **107**, 452 (1915).

² Hann, *J. Wash. Acad. Sciences*, **14**, 79 (1924).

³ Hann and Spencer, *J. Am. Chem. Soc.*, **49**, 535 (1927).

⁴ Raiford and Stoesser, *ibid.*, **49**, 1077 (1927).

⁵ Raiford and Hilman, *ibid.*, **49**, 1571 (1927).

⁶ Hantzsch and Schwab, *Ber.*, **34**, 834 (1901).

Unsuccessful attempts were made to prepare derivatives of chlorovanillin with *o*- and *p*-nitro-anilines, 2,4-dichloro-aniline and trinitro-aniline.

Experimental

The general procedure adapted was to weigh out 3 g. of chlorovanillin and an equimolecular proportion of the desired amine, add 25 cc. of 95% alcohol (or more if necessary for complete solution), heat to boiling and allow to digest at boiling temperature for two to two and one-half hours. Upon cooling, the reaction product usually separated in crystalline condition, or if an oil it could be induced to crystallize by scratching with a glass rod. The crude reaction product was recrystallized from alcohol to constant melting point, dried and analyzed. All melting points were taken with Anschütz thermometers and stems totally immersed in the heating bath. Table I gives a summary of the results.

TABLE I
SCHIFF'S BASES DERIVED FROM 5-CHLOROVANILLIN

Name	Formula	Appearance	M. p., (corr.), °C.	Analysis (Kjeldahl-Gunning-Arnold Method) 0.1 N HCl Nitrogen, %			
				Wt., g.	cc. consumed,	Found	Calcd.
5-Chlorovanillal <i>o</i> -toluidine	$C_{15}H_{14}O_2NCI$	Almost colorless cryst. powder	115	0.1022	3.5	4.80	5.10
5-Chlorovanillal <i>p</i> -toluidine	$C_{15}H_{14}O_2NCI$	Canary-yellow glisten- ing leaflets	142	.1125	4.0	4.98	5.10
5-Chlorovanillal <i>m</i> -nitro-aniline	$C_{14}H_{11}O_4N_2CI$	Light yellow powder	160	.1141	7.4	9.08	9.14
5-Chlorovanillal <i>p</i> -chloro-aniline	$C_{14}H_{11}O_2NCI_2$	Orange-yellow cryst. powder; light yel- low with gentle heat	128	.1548	4.8	4.34	4.73
5-Chlorovanillal cymidine	$C_{18}H_{20}O_2NCI$	Sl. yellow glistening truncated prisms	146-147	.1068	3.4	4.46	4.41
5-Chlorovanillal <i>p</i> -anisidine	$C_{15}H_{13}O_2NCI$	Brilliant straw-colored needle-like crystals	131	.2352	7.9	4.70	4.80
5-Chlorovanillal benzidine	$C_{28}H_{22}O_4N_2Cl_2$	Yellow powder	251-252	.1041	4.1	5.52	5.38
5-Chlorovanillal <i>m</i> -aminobenzoic acid	$C_{15}H_{12}O_4NCI$	Yellow hard cryst. crust	207	.1541	5.2	4.73	4.58
5-Chlorovanillal <i>p</i> -aminophenol	$C_{14}H_{12}O_3NCI$	Iridescent brick-red granules	150	.1229	4.3	4.90	5.05
5-Chlorovanillal <i>o</i> -dianisidine	$C_{22}H_{21}O_4N_2Cl_2$	Orange powder	188	.1226	5.8	6.63	6.79

The picrates of the Schiff's bases crystallize readily from alcoholic solutions and it is sometimes possible to obtain the picric acid addition product when the base itself cannot be isolated. In the present study the picrates were obtained by dissolving one gram of the pure base in 10 cc. of 10% alcoholic picric acid, heating to boiling and then allowing to cool, when the picrate separated. It was usually pure, but was recrystallized once from 95% alcohol before analysis. The results are given in Table II.

TABLE II
PICRATES OF SCHIFF'S BASES DERIVED FROM 5-CHLOROVANILLIN

Picrate of	Formula	Appearance	M. p., (corr.), °C.	Analysis			
				Salicyl Wt., g.	sulfonic consumed, cc.	acid method 0.1 N HCl Nitrogen, % Found	Calcd.
5-Chlorovanillal <i>m</i> -toluidine	$C_{21}H_{17}O_9N_4Cl$	Soft granular yellow micro-crystals	224	0.1237	9.8	11.10	11.10
5-Chlorovanillal <i>p</i> -toluidine	$C_{21}H_{17}O_9N_4Cl$	Soft brilliant golden yellow needles	230	.1206	9.3	10.80	11.10
5-Chlorovanillal <i>m</i> -nitro-aniline	$C_{20}H_{14}O_{11}N_5Cl$	Orange iridescent cryst. crusts	190	.1062	9.6	12.66	13.08
5-Chlorovanillal <i>p</i> -chloro-aniline	$C_{20}H_{14}O_9N_4Cl$	Orange-yellow granules	215	.1298	10.0	10.79	10.67
5-Chlorovanillal <i>p</i> -anisidine	$C_{21}H_{17}O_{10}N_4Cl$	Bright orange needles	229-230	.1188	9.1	10.73	10.76
5-Chlorovanillal benzidine	$C_{40}H_{28}O_{18}N_8Cl_2$	Micro-cryst. fine orange powder	250-260 (dec.)	.1660	13.3	11.22	11.44
5-Chlorovanillal ^a <i>m</i> -aminobenzoic acid alcoholate	$C_{21}H_{15}O_{11}N_4Cl \cdot$ C_2H_5OH	Orange-red hard granules	241	.1079 .2036 g. lost g. at 120°	7.4 .0170	9.61 8.35	9.65 7.93
5-Chlorovanillal <i>p</i> -aminophenol	$C_{20}H_{15}O_{10}N_4Cl$	Orange needles	224-225 (dec.)	.1712	13.2	10.80	11.06
<i>o</i> -Dianisidine	$C_{26}H_{22}O_{18}N_8Cl_2$	Soft golden yellow needles	Darkens, dec. 225°	.1018	11.4	15.69	15.96
5-Chlorovanillal <i>m</i> -aminobenzoic acid	$C_{21}H_{15}O_{11}N_4Cl$	Orange-yellow powder	236	.1144	8.5	10.41	10.48

^a Dried at 120° for five hours.

5-Chlorovanillal-*bis*-nitro-*p*-toluidine.—Five g. of nitrotoluidine (1-CH₃-2-NO₂-4-NH₂) and 6.1 g. of pure chlorovanillin were dissolved in 25 cc. of absolute alcohol and the solution was refluxed for two and one-half hours. Upon standing overnight in the ice box, the reaction mixture solidified to a mass of orange-yellow crystals. These were recrystallized twice from 95% alcohol, from which the compound separated in hard yellow flower-like crystalline rosetts which melted at 125° (corr.) to a clear yellow oil. Although the experiment was repeated three times no compound formed by a condensation of a single amine molecule could be isolated.

The picrate of the *bis*-compound separated in bright yellow micro-crystals when one gram of base was treated with 10 cc. of 10% alcoholic picric acid. It melted, although not sharply, at 148° to a clear red oil.

Anal. Base, subs., 0.1697: 0.1 N HCl, 14.1 cc. Calcd. for C₂₂H₂₁O₆N₄Cl, 11.85. Found: 11.64. Picrate, subs., 0.2019: 0.1 N HCl, 20.1 cc. Calcd. for C₂₈H₂₄O₁₃N₇Cl: N, 13.97. Found: N, 13.94.

Summary

Chlorovanillin has been condensed with *o*-, *m*- and *p*-toluidines, *m*-nitro-aniline, *p*-chloro-aniline, cymidine, *p*-anisidine, nitro-*p*-toluidine, benzidine, *m*-aminobenzoic acid, *p*-aminophenol and dianisidine, and the condensation products have been characterized by the preparation of their addition products with picric acid.

Phenacyl, *p*-Chlorophenacyl and *p*-Bromophenacyl Esters of Some Higher Fatty Acids*

The utilization of phenacyl and para halogen phenacyl esters for the identification and separation of acids has been indicated by Rather and Reid¹ and by Judefind and Reid.² The successful employment of these reagents for fruit acids has suggested the possibility of quantitative differentiation of the fatty acids obtained from the saponification of the glycerides contained in fats and vegetable oils. The present accepted analytical procedure for substances of this nature consists in esterifying the acids obtained upon saponification, followed by repeated fractionation in vacuo, resaponification and recovery and identification of the free acids. This process, while quantitatively exact, is time consuming and requires close attention and care for its successful completion.

Unfortunately the solubilities of the phenacyl and halogen phenacyl esters of the higher fatty acids are of such an order as to preclude their use in this regard. They are, however, beautiful crystalline compounds which are of value for the establishment of the identity of the higher members of the fatty acid series.

TABLE I
PROPERTIES OF ESTERS OF SOME HIGHER FATTY ACIDS

Ester of	M. p., °C.	Appearance	Soly., g. in 100 cc. of 95% EtOH	
			20°	25°
Phenacyl Esters				
Lauric	48-49	Long, soft acicular needles	2.9150	5.3800
Myristic	56	Soft fluffy plates	1.6980	1.7490
Palmitic	63	Brilliant flat scales	0.5136	0.7890
Stearic	69	Dull cottony needles	.2160	.3650
Arachidic	85-86	Soft white powder	.1348	.2660
Lignoceric	87-88	Soft white powder	.1204	.1816
<i>p</i> -Chlorophenacyl Esters				
Lauric	70	Brilliant scales	0.6060	0.7856
Myristic	76	Brilliant glistening scales	.2472	.3071
Palmitic	82	Brilliant soft needles	.0784	.1020
Stearic	86	Microcrystalline felted needles	.0648	.1000
Arachidic	86	Microcrystalline aggregates	.0100	.0125
Lignoceric	99-100	Microcrystalline powder	.0054	.0072
<i>p</i> -Bromophenacyl Esters				
Lauric	76	Brilliant scales	0.3832	0.4288
Myristic	81	Brilliant platelets	.1600	.2092
Palmitic	86	Brilliant scales	.0512	.0684
Stearic	90	Brilliant platelets	.0200	.0260
Arachidic	89	Dull microcrystalline aggregates	.0080	.0096
Lignoceric	90-91	Dull microcrystalline powder	.0040	.0072

* Hann, Reid and Jamieson, *J. Am. Chem. Soc.*, **52**, 818 (1930)

¹ Rather and Reid, *ibid.*, **41**, 75 (1919).

² Judefind and Reid, *ibid.*, **42**, 1043 (1920).

Experimental

The acetophenones and phenacyl bromides were prepared in accordance with the directions of Judefind and Reid. In the preparation of *p*-chloro-acetophenone an appreciable quantity of *p*-chlorobenzoic acid was isolated as a secondary reaction product. In brominating *p*-chloro-acetophenone, it was found convenient to dissolve it in twice its weight of glacial acetic acid, add exactly one molecular quantity of bromine, heat gently to decolorization and cool in ice. Pure ω -bromo-*p*-chloro-acetophenone then separated in brilliant plates in quantitative yield.

The general method of procedure adopted for the preparation of the esters was as follows. One gram of acid was weighed out, an amount of normal sodium hydroxide almost sufficient to neutralize the acid was added, and the mixture was boiled with 10 cc. of 95% alcohol until acid to phenolphthalein. An excess of acid is essential to prevent formation of the corresponding phenacyl alcohol, which would contaminate the crystalline fatty acid ester. A molecular proportion of the desired phenacyl or halogen phenacyl bromide was then introduced, and the reaction mixture boiled under a reflux condenser with the addition of sufficient alcohol to keep the reaction product in solution. After being heated for one hour, the flask was cooled, and the separated solid recrystallized from dilute alcohol to constant melting point. Analysis for halogen by the Parr bomb proved the compounds to be the expected esters.

All the phenacyl compounds prepared are colorless crystalline solids, the majority of them exhibiting a marked luster. This property decreases with increase in molecular weight, and a tendency is noted toward decrease in crystal size with increase in molecular weight.

Approximate solubility determinations were made by removing a 25-cc. portion of the saturated solution from solutions maintained at 20 and 25° in suitable constant temperature baths and drying to constant weight.

The data which have been accumulated are included in Table I.

Summary

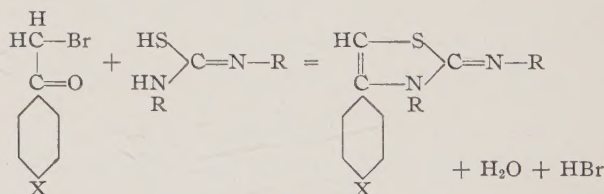
Phenacyl, *p*-chlorophenacyl and *p*-bromophenacyl esters of lauric, myristic, palmitic, stearic, arachidic and lignoceric acids have been prepared and described.

Isomeric 2-Iminotolyl-3-tolyl-4-*p*-chloro and *p*-Bromophenyl- Δ^4 -thiazolines*

In continuation of a series of researches upon the relation between constitution and color absorption of certain heterocyclic compounds, advantage has been taken of the reactive nature of the ω -halogen acetophenones in condensation reactions with symmetrically disubstituted thioureas to prepare a series of iminoarylthiazolines.

Hantzsch¹ in 1888 published a paper which not only outlined the complete field of the azoles, but was a masterpiece of scientific recapitulation of the particular group in which he was then interested, namely, the thiazoles and their reduction products. This résumé in part reported the pioneer work of Traumann,² one of his pupils, who investigated the action of halogen ketones upon thiourea, monoalkyl thioureas and symmetrical dialkyl thioureas. The general utilization of aryl ketones and thioureas was introduced by von Walther,³ who advanced a theory regarding the mechanism of the condensation reaction involved.

The present paper details the application of the *p*-halogen-phenyl ω -bromomethyl ketones as synthetic agents in the preparation of some isomeric 2-iminotolyl-3-tolyl-4-*p*-halogen-phenyl- Δ^4 -thiazolines. The general reaction is



Since the iminothiazolines are strong bases, the halogen acid released during the condensation is reabsorbed to yield the hydrobromide of the base. In the present study no effort was made to isolate the intermediate compounds, but the free base was obtained directly by alkalization of the reaction mixture. The bases were characterized, however, by preparation of the addition compounds with picric acid.

Experimental

The ω -bromoacetophenone was prepared as described by Rather and Reid⁴ and the para halogenated compounds according to the general procedure of Judefind and Reid.⁵

* Hann and Reid, *J. Am. Chem. Soc.*, **55**, 4998 (1933).

¹ Hantzsch, *Ann.*, **249**, 1 (1888).

² Traumann, *ibid.*, **249**, 31 (1888).

³ Von Walther, *J. prakt. Chem.*, [2] **75**, 187 (1907).

⁴ Rather and Reid, *J. Am. Chem. Soc.*, **41**, 77 (1919).

⁵ Judefind and Reid, *ibid.*, **42**, 1044 (1920).

The bromination of *p*-chloroacetophenone was conducted as indicated by Hann, Jamieson and Reid⁶ in their study of the phenacyl esters of the fatty acids. The thioureas were obtained by the interaction of mustard oils with the required amine.

The condensation of the *p*-*o*-dihalogen ketones with the thioureas was effected by heating molecular proportions of the constituents in absolute alcohol for a period of two hours. In certain cases separation of the thiazoline hydrobromide took place before the expiration of this time period, in which case solution was effected by addition of more alcohol. To the alcoholic solution, which in a majority of cases could be cooled without solid separating, was now added 100 cc. of normal sodium hydroxide and the free thiazoline base precipitated as a gum or crystalline solid. This was washed with water and recrystallized from alcohol or acetone until physical properties were constant. Analyses were conducted by the Kjeldahl-Gunning-Arnold procedure and melting points were obtained with Anschütz thermometers with stems totally immersed. The yields were 80 to 90%. The data are summarized in Table I.

TABLE I

ISOMERIC 2-IMINOTOLYL-3-TOLYL-4-*p*-HALOGEN-PHENYL- $\Delta^{4,5}$ -THIAZOLINES

Compound, phenyl- $\Delta^{4,5}$ -thiazoline	Appearance
1 2-Iminophenyl-3-phenyl-4- <i>p</i> -chloro- ^a	Colorless micro-crystalline needles
2 2-Iminophenyl-3-phenyl-4- <i>p</i> -bromo-	Colorless micro-crystalline needles
3 2-Imino- <i>o</i> -tolyl-3- <i>o</i> -tolyl-4- <i>p</i> -chloro-	Slightly yellow brilliant granules
4 2-Imino- <i>o</i> -tolyl-3- <i>o</i> -tolyl-4- <i>p</i> -bromo-	Slightly yellow brilliant crystalline aggregates
5 2-Imino- <i>p</i> -tolyl-3- <i>p</i> -tolyl-4- <i>p</i> -chloro-	Colorless opaque micro-crystalline groups
6 2-Imino- <i>p</i> -tolyl-3- <i>p</i> -tolyl-4- <i>p</i> -bromo-	Opaque colorless asbestos-like shreds

^a The hydrochloride of this base separates in colorless acicular needles melting at 228–229°C. (corr.).

Formula	M. p., °C. (corr.)	Analyses, Kjeldahl-Gunning Arnold Method			
		Weight, g.	Cc. of 1 N HCl consumed	Nitrogen, % Found	Calcd.
1 C ₂₁ H ₁₅ N ₂ SCl	204	0.1347	7.3	7.59	7.72
2 C ₂₁ H ₁₅ N ₂ SBr	206	.1015	5.0	6.90	6.88
3 C ₂₃ H ₁₉ N ₂ SCl	132	.1255	6.3	7.03	7.17
4 C ₂₃ H ₁₉ N ₂ SBr	123	.1058	4.9	6.49	6.44
5 C ₂₃ H ₁₉ N ₂ SCl	227	.1038	5.2	7.02	7.17
6 C ₂₃ H ₁₉ N ₂ SBr	239	.1367	6.0	6.15	6.44

The basic character of the thiazolines is emphasized by the ease of reaction with picric acid to form beautifully crystalline picrates. One gram of base was dissolved in a minimum amount of alcohol and 10 cc. of alcoholic 10% picric acid added. An immediate separation of the picrate usually occurred and in a number of instances the solution set to a solid mass due to the interwoven structure of the picrate crystals. The picrates were filtered by suction, washed with 95% alcohol and recrystallized from alcohol. Analyses were conducted in this case by the salicyl-sulfonic acid method in order to avoid loss of nitro nitrogen. The description and physical properties of the compounds prepared are included in Table II.

⁶ Hann, Jamieson and Reid, *J. Am. Chem. Soc.*, **52**, 818 (1930).

TABLE II

PICRATES OF ISOMERIC 2-IMINOTOLYL-3-TOLYL-4-*p*-HALOGEN-PHENYL- $\Delta^{4,5}$ -THIAZOLINES

Picrates of Δ^4 -thiazoline		Appearance
1	2-Iminophenyl-3-phenyl-4- <i>p</i> -chlorophenyl-	Golden shimmering platelets
2	2-Iminophenyl-3-phenyl-4- <i>p</i> -bromophenyl-	Golden shimmering platelets
3	2-Imino- <i>o</i> -tolyl-3- <i>o</i> -tolyl-4- <i>p</i> -chlorophenyl-	Yellow cleavage-like crystals
4	2-Imino- <i>o</i> -tolyl-3- <i>o</i> -tolyl-4- <i>p</i> -bromophenyl-	Yellow brilliant aggregates
5	2-Imino- <i>p</i> -tolyl-3- <i>p</i> -tolyl-4- <i>p</i> -chlorophenyl-	Yellow brilliant needles
6	2-Imino- <i>p</i> -tolyl-3- <i>p</i> -tolyl-4- <i>p</i> -bromophenyl-	Yellow lustrous granules

Analyses, salicyl sulfonic acid method						
Formula	M. p., °C. (corr.)	Weight, g.	Cc. of 1 N HCl consumed	Nitrogen, %		
				Found	Calcd.	
1 C ₂₇ H ₁₈ O ₇ N ₆ SCl	206	0.1331	11.0	11.58	11.84	
2 C ₂₇ H ₁₈ O ₇ N ₆ SBr	203	.1209	9.4	10.89	11.01	
3 C ₂₉ H ₂₂ O ₇ N ₆ SCl	187	.1237	9.7	10.98	11.30	
4 C ₂₉ H ₂₂ O ₇ N ₆ SBr	196	.1145	8.5	10.40	10.54	
5 C ₂₉ H ₂₂ O ₇ N ₆ SCl	183	.1115	9.0	11.31	11.30	
6 C ₂₉ H ₂₂ O ₇ N ₆ SBr	193	.1713	12.6	10.30	10.54	

Summary

p-Chloro and *p*-bromo- ω -acetophenones have been condensed with diphenyl, di-*o*-tolyl and di-*p*-tolyl thioureas to yield 2-imino aryl-3-aryl-4-*p*-halogen-phenyl- Δ^4 -thiazolines. The corresponding picrates have also been isolated and described.

VITA

Raymond M. Hann was born January 28, 1900, at Washington, D. C., the son of Alvah Rittenhouse and Mary Regina Hann. He attended the primary and secondary schools of Washington and received the B.S. degree (with distinction) from the George Washington University in 1925 and the M.S. degree from the same institution in 1926. The degree of Doctor of Philosophy with organic chemistry as the principal subject was granted by the Johns Hopkins University in 1928.

He was employed by the Bureau of Chemistry from 1918 until 1927 except for a short period during the war. He held a research fellowship at the Mellon Institute of Industrial Research from 1927 to 1929, resigning to enter the service of the National Institute of Health.

He is the author of some thirty papers covering various fields of organic chemistry particularly in studies of the heterocyclic thiazoles.

